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APPLICATION NUMBER: 60/555,427

FILING DATE: *March 23, 2004*

RELATED PCT APPLICATION NUMBER: *PCT/US05/09726*



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U.S. PTO

PROVISIONAL APPLICATION COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION under 37 CFR 1.53 (c).

Express Mail No.: EV 044751207 US

17613 60/555427 U.S.PTO



MAIL STOP PROVISIONAL APPLICATION
 Commissioner for Patents
 P.O. Box 1450
 Alexandria, VA 22313-1450

Docket Number: BU-120Xq800

Type a Plus sign (+)
inside this box → +

INVENTOR(s)/APPLICANT(s)

LAST NAME	FIRST NAME	MIDDLE INITIAL	RESIDENCE (CITY AND EITHER STATE OR FOREIGN COUNTRY)
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Murray	Todd	W.	Brookline, Massachusetts
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[] Additional Inventors are being named on Page 2 attached.
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TITLE OF THE INVENTION (280 characters max)

DEVICE AND METHOD FOR HIGH SENSITIVITY LASER ULTRASONIC
 CHARACTERIZATION OF MICRO- AND NANOSCALE MATERIALS

CORRESPONDENCE ADDRESS

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ENCLOSED APPLICATION PARTS (CHECK ALL THAT APPLY)

[X] Specification Number of pages [29]	[X] Small Entity status is entitled to be, and hereby is, asserted for this application
[] Drawing(s) Number of sheets []	[] Other (specify)

METHOD OF PAYMENT (CHECK ONE)

[X] A check in the amount of \$80.00 is enclosed to cover the Provisional Filing Fee
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[] The Commissioner is hereby authorized to charge filing fees and credit Deposit Account Number 23-0804

Please recognize the following attorneys with powers in this application.

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SIGNATURE

DATE

3-23-4

TYPED or PRINTED NAME: Charles L. Gagnebin III
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U.S. PROVISIONAL APPLICATION

ENTITLED

**DEVICE AND METHOD FOR HIGH SENSITIVITY LASER ULTRASONIC
CHARACTERIZATION OF MICRO- AND NANOSCALE MATERIALS**

BY

TODD W. MURRAY

EXPRESS MAIL NO: EV 044751207 US

Describe the invention, with the following points:

a. General purpose of invention;

The general purpose of the invention is the nondestructive evaluation of thin films and coating materials to determine the mechanical and thermal properties of these materials, thickness, or to identify flaws or defects in material structures.

b. Technical description of invention;

The laser-based ultrasonic system uses a high frequency modulated laser diode source which is amplified using a fiber optic amplifier to generate narrow-band acoustic waves in materials at frequencies up to tens of GHz or more. The generation frequency is scanned through the frequency range of interest and the signals are detected using a standard interferometry system coupled to a sensitive lock-in detection scheme. The signals may then be processed in the frequency domain or time domain signals may be reconstructed from the measured magnitude and phase signals, allowing for time domain "gating" to identify the acoustic modes or arrivals of interest. The latter approach is beneficial in cases where the vibrational modes of the entire structure under inspection contribute to the background noise of the detection system. The physical properties of the micro- or nanoscale structure (ie. thin films) are determined through analysis of the measured acoustic data. The system as described above can also be used for high sensitivity evaluation of thermal properties of micro- and nanoscale materials.

c. Advantages and improvements over existing methods, devices or materials;

The modulated laser based film inspection system has extremely high sensitivity due to the narrow bandwidth over which the measurements are made. This is a substantial improvement over existing systems which use pulsed laser sources. The improvements in sensitivity also allow for film measurements to be made with lower incident power and thus with lower heating of the target material and less chance of target material damage. In addition, GHz modulated laser sources are substantially less expensive and more compact than femtosecond sources used in existing systems.

d. Possible variations and modifications;

e. Features believed to be new;

The system provides unprecedented sensitivity through the use of a lock-in detection scheme. The characterization of micro- and nanoscale materials requires high frequency acoustic wave generation and this has been accomplished in existing systems through the use of picosecond or femtosecond laser sources. Several existing patents cover these areas. This system uses a low power GHz modulated laser source to generate low amplitude (fermtometer) acoustic waves. In the proposed system, the bandwidth can be reduced simply by increasing the time constant on the lock-in allowing us to control the signal to noise ratio. The system uses low power (compared to pulsed systems) laser sources which lead to very little surface heating thus limiting the chance of thermally induced changes in the sample surface during the inspection procedure. The ability to reconstruct the time domain waveforms to obtain data similar to that obtained using pulsed systems allows for time gating of the desired acoustic mode.

f. Close or related patents (if known);

g. Problem solved (if applicable);

h. Possible uses of invention ;

Determining the physical and mechanical properties of micro- and nanoscale systems, defect detection, thermal characterization.

i. Disadvantages or limitations;

i. Non confidential description;

A novel technique has been developed to measure the physical and mechanical properties of micro- and nanoscale systems in a non-contact and nondestructive manner using laser sources.

Addition Details for BU disclosure/Patent Application
Todd Murray

Title:

Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro- and Nanoscale Materials

Conventional laser ultrasonic techniques for the characterization of micro and nanoscale structures (including films and coatings) use pulsed laser sources for the generation of acoustic waves [1-3]. Short laser pulses from femtosecond and picosecond lasers have high peak powers and generate broad-bandwidth acoustic waves. In this disclosure, a new technique for acoustic microscopy using high frequency modulated continuous wave (CW) laser sources is presented. This technique is suitable for:

- a) Generation and detection of acoustic waves in films and coating materials for nondestructive evaluation and determination of physical and mechanical properties.
- b) Generation and detection of resonant vibrations in nanometer electromechanical systems (NEMs) and micro-electromechanical systems (MEMs) for nondestructive evaluation and determination of mechanical properties. Examples include NEMs and MEMs components such as cantilever beams and membranes.
- c) Generation and detection of acoustic waves in macroscopic systems for nondestructive evaluation and determination of physical and mechanical properties. Applications also include the detection of subsurface or surface breaking cracks, and the detection of subsurface voids or disbands.

Recent developments in laser technology, geared primarily for the telecom sector, have resulted in electro-absorption modulated DFB diode laser sources that can be amplitude modulated at frequencies approaching 40GHz. In addition, erbium doped fiber amplifiers are available to amplify the output of these lasers. It is noted that these components are relatively inexpensive, especially when compared to femtosecond and picosecond solid state laser systems. The result is high power laser sources that can be modulated at GHz frequencies. A new technology for acoustic microscopy has been developed which makes use of these lasers to develop a high resolution CW photoacoustic microscopy system with the potential to operate at frequencies approaching 40 GHz.

The reasons for exploring photoacoustic microscopy using a CW laser source are twofold. First, it is expected that the signal-to-noise ratio of a CW system will be *significantly improved* over that of a pulsed system in a number of cases. Next, it is possible to resonantly excite small scale structures such as coatings, membranes, beams used in MEMs applications using a CW laser source to evaluate their mechanical properties. Consider that for thermoelastic generation of acoustic waves, there exists some temperature T_{max} (typically taken as the melting point)

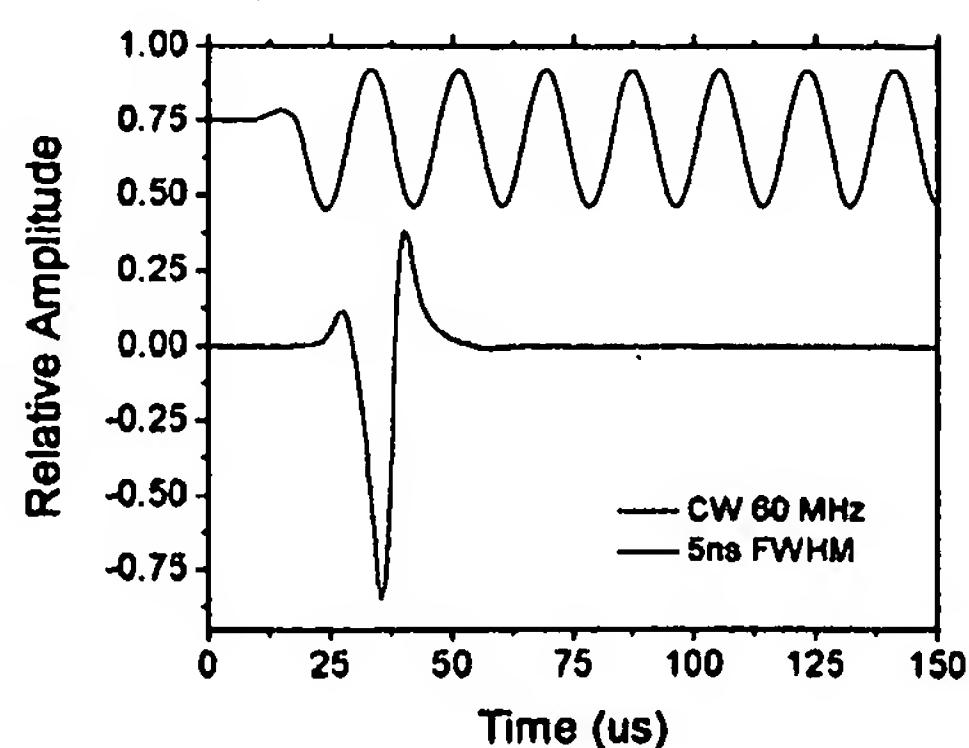


Figure 1. Acoustic response of an aluminum half-space showing SAWs generated using CW and pulsed laser sources. The laser sources produce the same maximum surface temperature in the sample.

that the sample surface is kept below in order to avoid damage or ablation. For a given laser pulse shape, this limits the maximum allowable absorbed power density at the surface. Laser heating with a 5ns Gaussian laser pulse is compared with that produced by a 60 MHz CW laser source. The laser spot size is taken as $3\mu\text{m}$. It is found that, for the same absorbed power density in each case, the CW laser heats the material to a temperature of approximately 2.5 times higher than the pulsed laser. This is due to the fact that heat builds up in the sample between cycles until the sample reaches steady state. Now, the CW laser power is scaled down by a factor of 2.5 such that both of the laser sources produce *equivalent surface heating*. The scaled pulse shapes are then convolved with the impulse response of an aluminum semi-infinite half space (with the source and receiver slightly offset on the sample surface) to find the acoustic response of the sample. The resulting signals are shown in Figure 1. As is evident in the pulsed laser case, the laser source produces a strong surface acoustic wave (SAW). For laser powers that produce equivalent surface heating, the SAW displacement amplitude is a factor of about 2.5 higher than that of CW generation, but the bandwidth of the CW signal can be substantially reduced through detection with an RF lock-in amplifier. Using a sufficiently long integration time the bandwidth can be reduced by six orders of magnitude for the narrowband case over the broadband case resulting in a *SNR increase of three orders of magnitude* for this particular example. SNR is an important issue in laser based systems, which have substantially lower sensitivity than conventional contact transducers [1], and this type of SNR increase could open up the possibility of using these non-contact systems for a much wider range of inspection applications. We note that the improved SNR is strongly dependent on the spot size and thermal conductivity of the specimen. For laser ultrasonic microscopy, though, small spot sizes are required to produce localized sources and to make measurements over short source to receiver distances. It is also noted that the above analysis assumes that a high power CW laser source, capable of operating just below the ablation threshold of the sample, is available. Using diffraction limited spot sizes in the $0.75\text{-}3.0\mu\text{m}$ range, peak power densities in the 100's of MW/cm^2 can be achieved with CW lasers operating in the standard 1-10W range.

One of the disadvantages of CW generation is that the signals can be difficult to interpret, especially when multiple acoustic arrivals are present. Two approaches are proposed to circumvent this difficulty.

- a) Time domain signals are reconstructed through the measured magnitude and phase over a large bandwidth. This is done using, for example, a vector network analyzer or lock-in amplifier and inverting the frequency domain data. The frequency domain data (magnitude and phase) are collected at each frequency and the frequency domain data converted to the time domain using an inverse Fourier Transform. This time domain data can then be time gated to extract the signal of interest. Note that in principle the frequency bandwidth is only limited by the maximum modulation frequency of the source.
- b) Next, pulse coding techniques such as using a linear FM or chirped generation pulse and pulse compression (matched filtering) can also be used to obtain high temporal resolution.

The second reason for using CW generation, especially at high frequencies, is that it opens up the possibility of doing true acoustic spectroscopy. The first longitudinal thickness resonance in a $1\mu\text{m}$ thick aluminum film, for instance, is at around 3 GHz. By scanning the frequency and observing the amplitude and phase of the material response, extremely precise measurement of acoustic wave velocities are possible.

A basic schematic of one possible mode of implementation of the invention is shown in Figure 2. The system has three optical paths that lead to the sample surface through the same microscope objective. The first path leads to a CCD camera and allows for high resolution optical imaging of the sample surface as well as sample alignment. In the second path, the detection laser light enters the microscope though a single mode optical fiber, is collimated, and directed to the sample surface. Upon reflection from the sample the light is returned to a stabilized Michelson interferometer where the acoustic signal of interest is detected. The generation laser is collimated, directed to a mirror on a gimbal mount, sent though a relay lens system, and directed to the specimen. The gimbal mount allows for precise control of the generation point within the field of view of the microscope. An electroabsorption modulated DFB diode laser with a fiber amplifier is used for generation. Typical lasers can be modulated at frequencies exceeding 10 GHz. The output of the photodiode is sent to an RF lock-in amplifier for frequency domain measurements up to 200 MHz. For higher frequency measurements, the RF signal will be mixed down to the 0-200MHz frequency range using a phased locked oscillator and commercially available mixers covering the frequency range of interest.

Additional claims:

- a) An array of laser sources can be used for phased array generation and detection of acoustic waves providing a boost in signal to noise ratio and providing beam steering capability. The individual sources can be phased relative to one another through providing phase delays to the amplitude modulators.
- b) The invention can be used for high frequency biological imaging. Using a source laser of 1550nm, which is highly absorbed in water, acoustic waves can be launched into a liquid containing a biological media.
- c) Other techniques for amplitude modulation of the generation source including mach-zehnder or acousto-optic modulators may be used.

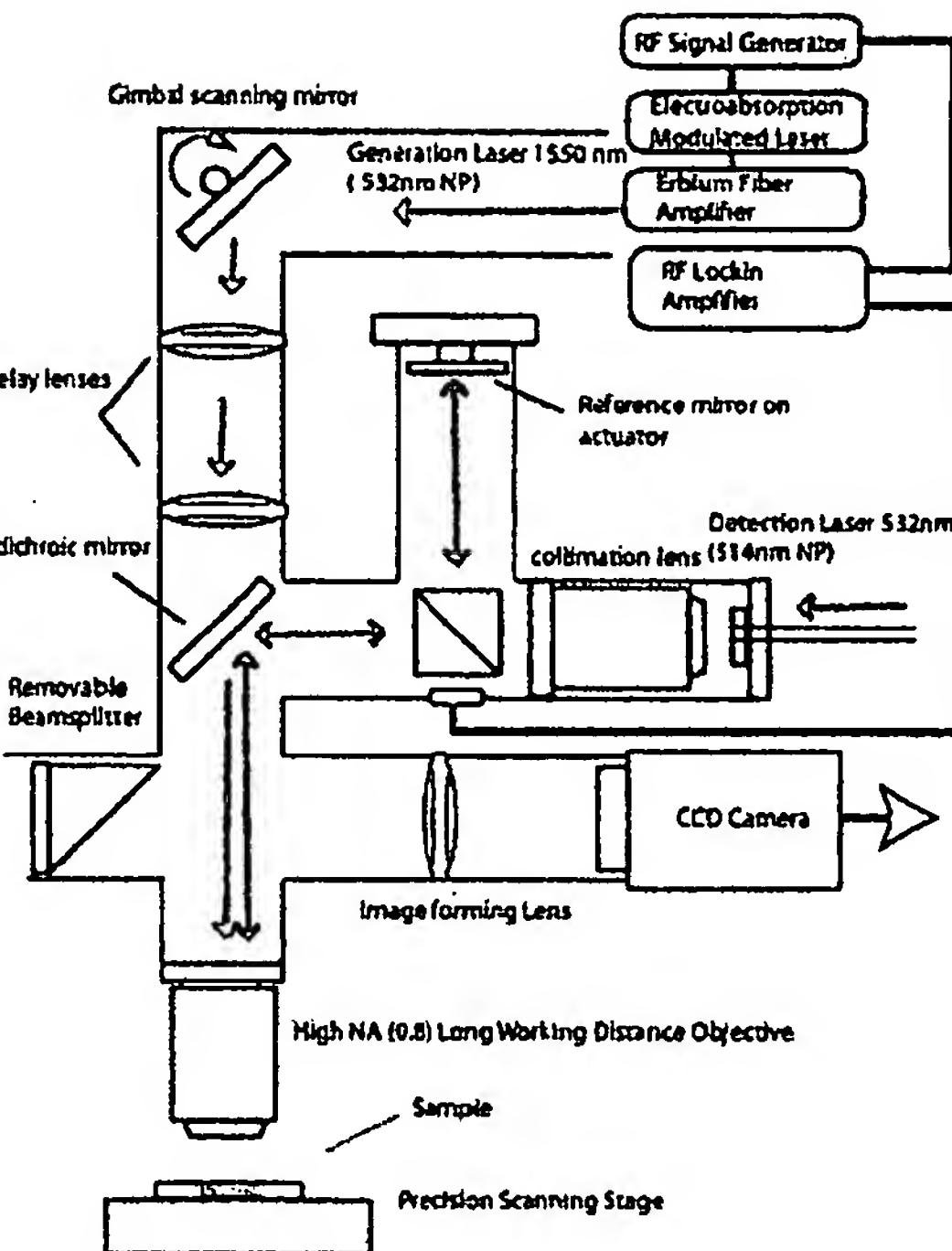


Figure 2. Schematic of high frequency, CW source modulation, acoustic microscopy system.

¹ C.B. Scruby and L.E. Drain, *Laser Ultrasonics, Techniques and Applications*, Adam Hilger, N.Y. (1990).

² S.J. Davies, C. Edwards, G.S. Taylor, and S.B. Palmer, "Laser-generated ultrasound: its properties, mechanisms, and multifarious applications," *J. Phys. D* 26 329-348 (1993).

³ J.P. Monchalin, C. Neron, J.F. Bussiere, P. Bouchard, C. Padoleau, R. Heon, M. Choquet, J.D. Aussel, G. Durou, and G. Nilson, "Laser-ultrasonics: From the laboratory to the shop floor," *Adv. Perform. Mater.* 5 (1-2) 7-23

Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials: T.W. Murray

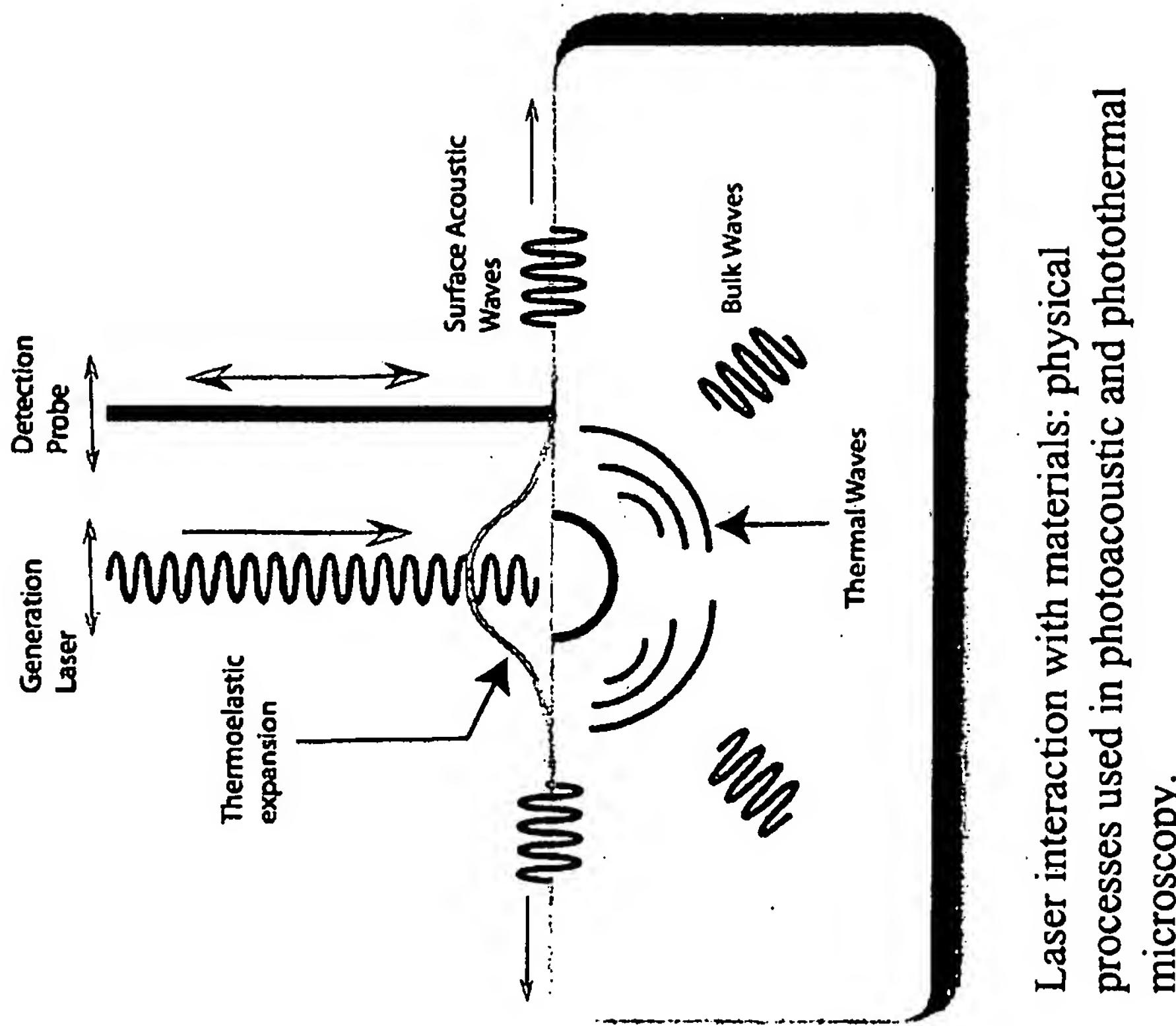
Photoacoustic Microscopy

- Pulsed or modulated CW lasers illuminate target surface
- Absorption of incident light leads to local surface heating
- Thermoelastic expansion launches acoustic waves
- Waves detected with optical probe
- Determine mechanical and thermal properties of target
- Detect defects in materials (voids, disbonds, microcracks)

∞

Advantages of Photoacoustic Microscopy

- non-contact, remote inspection
- extremely high fidelity (Hz-GHz)
- high spatial resolution
- rapid scanning possible
- well suited for the inspection of MEMS, NEMs, thin films and coatings



Laser interaction with materials: physical processes used in photoacoustic and photothermal microscopy.

Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

High Frequency Laser Acoustic Generation Schemes

- ☐ Picosecond and femtosecond pump probe techniques
- ☐ Conventional technique for high frequency laser ultrasonics
Bandwidth of ultrasound is typically in GHz range
- ☐ Impulse stimulated thermal scattering
Crossed laser pulses used to launch narrow-band acoustic signals: Bandwidth still relatively large if high spatial resolution required
Maris 1998, Richardson et al. 1999, Rogers et al., 2000

CW Modulated Laser Ultrasonic Systems

- ☐ Ultrasonic waves are generated at discrete frequencies using an amplitude modulated laser.
- ☐ Narrow bandwidth of the ultrasonic waves enhances the signal sensitivity by several orders of magnitude, compared to broadband techniques.
- ☐ Lower cost compared to broadband laser acoustic systems.
- ☐ Challenges
CW generated ultrasonic signals may contain multiple modes that can render signal interpretation difficult.

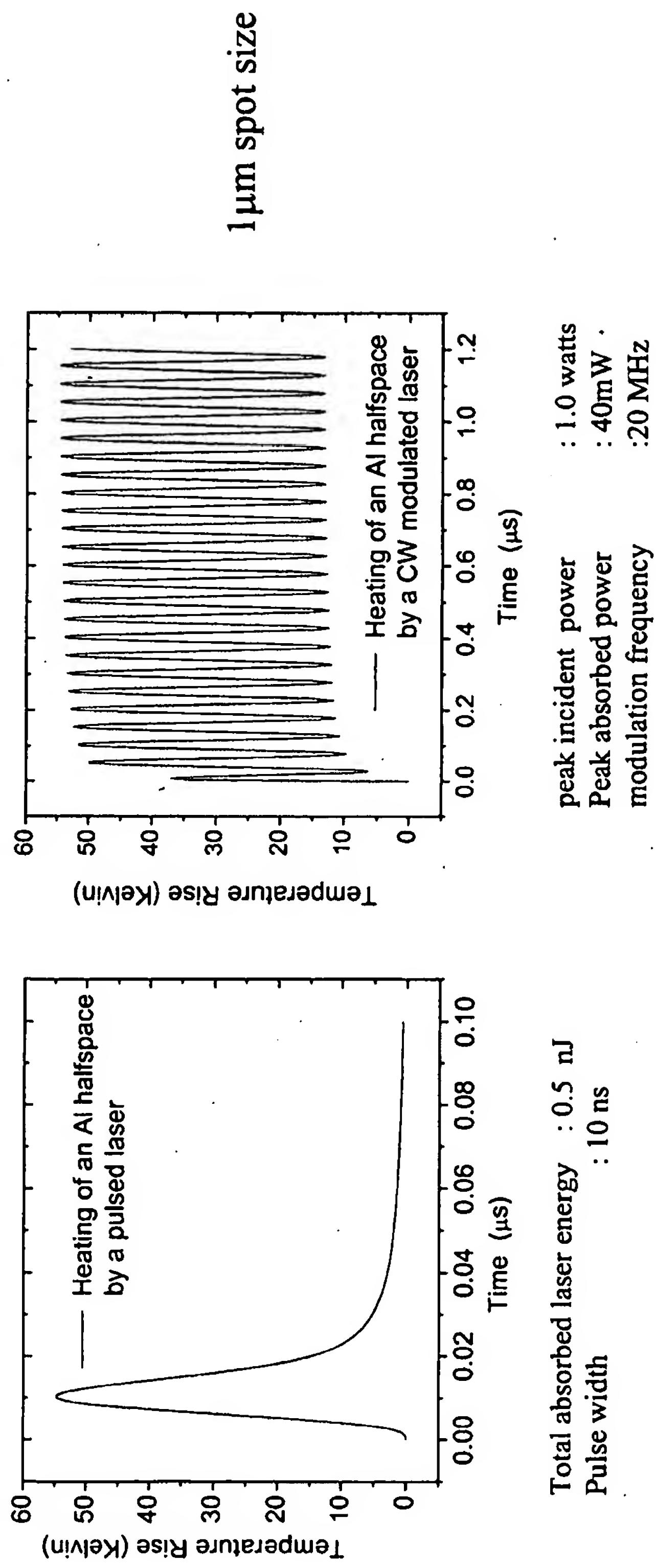
Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

High resolution photoacoustic/photothermal imaging requires small spot size: *ultimately we want to generate and detect acoustic waves with nearfield optical sources.*

How do we generate the acoustic signals?

If pulsed sources are used target ablation threshold severely limits the amount of laser energy that can be used/ low sensitivity limits the utility of this technique

o We have developed a high sensitivity technique for acoustic microscopy using an RF modulated diode laser source with time domain reconstruction.



Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

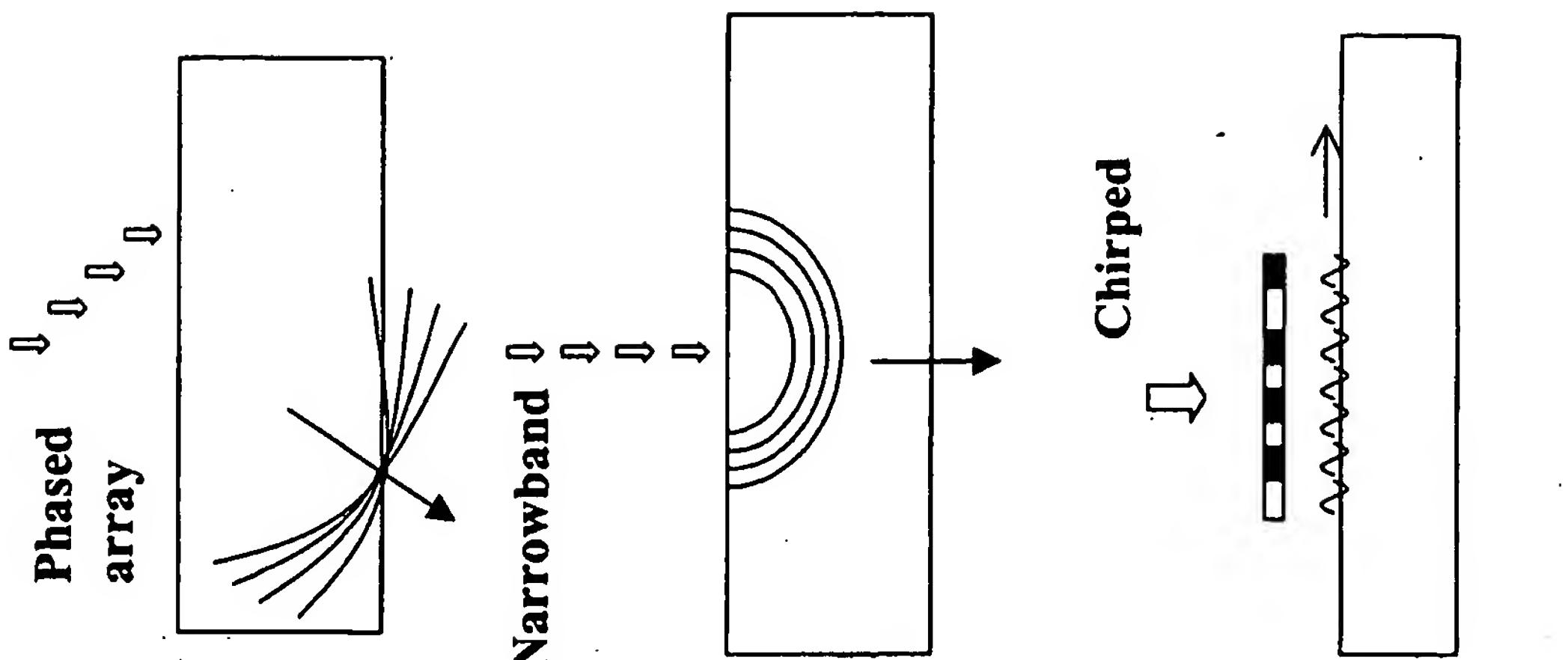
SNR of shot noise limited optical detection system:

$$\text{SNR} \propto \frac{\delta^2 P}{B}$$

δ - displacement

P- Optical power incident on detector

B- bandwidth of detection system



1. Multi-Pulse Methods

- use multiple laser sources to generate narrow bandwidth or phased acoustic pulses

2. Single Pulse Methods

- modify spatial profile of single laser pulse to optimize SNR (narrow-band/ chirped)

3. Ablation

- Is a small amount of surface damage acceptable for a given application ?

Chirped



1. T.W. Murray, J.B. Deaton Jr., and J.W. Wagner, "Experimental evaluation of enhanced generation of acoustic waves using an array of laser sources," *Ultrasonics* (1996)
2. J.S. Steckenrider, T.W. Murray, J.W. Wagner, and J.B. Deaton Jr., "Sensitivity enhancement in laser ultrasonics using a versatile laser array system," *J. Acoust. Soc. Am.* (1995)
3. T.W. Murray, K.C. Baldwin, and J.W. Wagner, "Laser ultrasonic chirp sources for low damage and high detectability without loss of temporal resolution," *J. Acoust. Soc. Am.* (1997)

Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

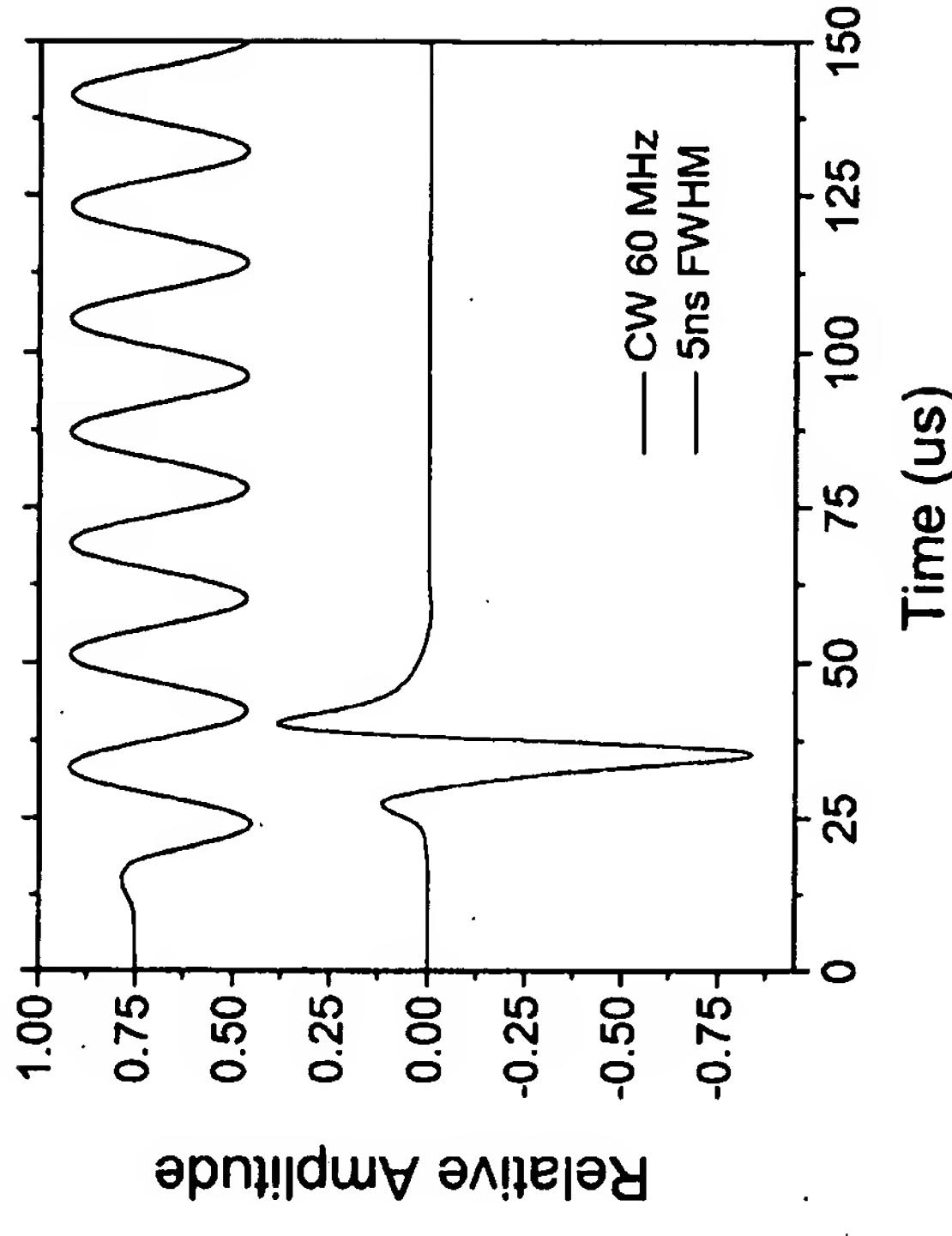
Basic idea of sensitivity enhancement through CW generation

$$T = \frac{F_{\max} d^2 \sqrt{k}}{K \sqrt{\pi}} \int \frac{p(t - t_1) dt_1}{\sqrt{t_1(4kt_1 + d^2)}} \exp\left(-\frac{z^2}{4kt_1} - \frac{r^2}{4kt_1 + d^2}\right)$$

All laser ultrasonic systems ultimately limited by
The ablation threshold of the target material

- calculate pulsed and CW laser power which will produce equal surface heating
- convolve each optical signal with impulse response of system
- compare resulting surface displacement

→



SNR of shot noise limited optical

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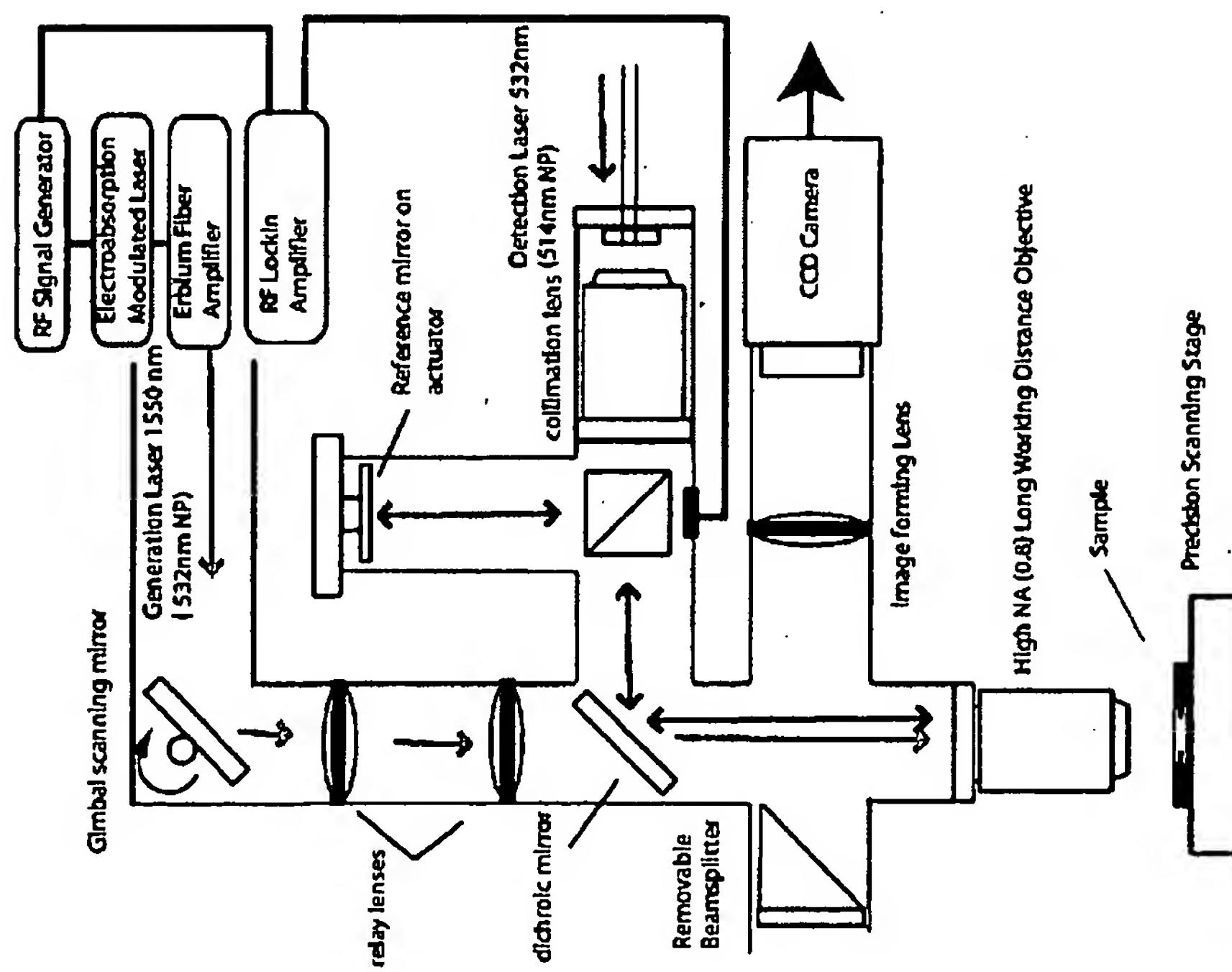
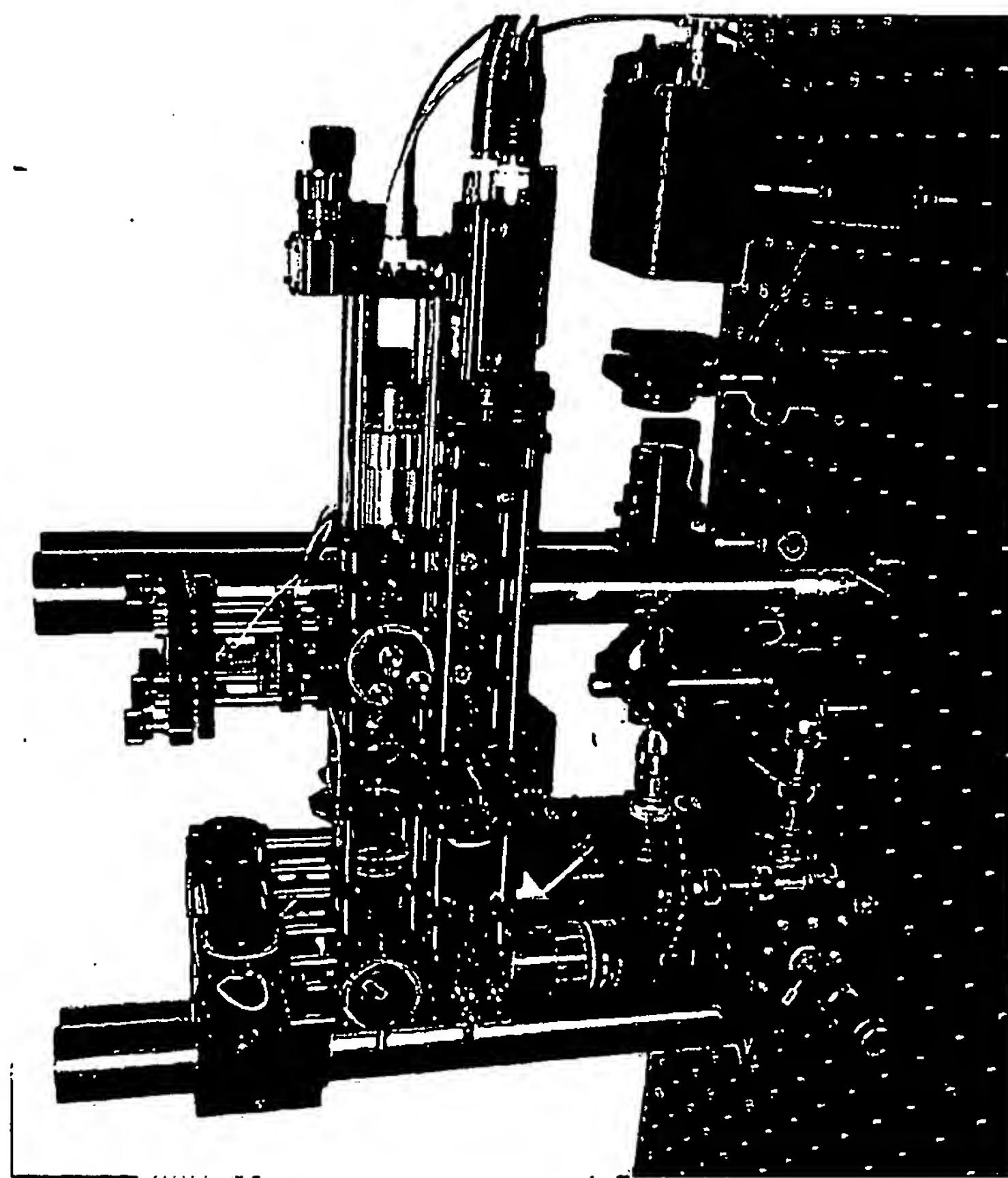
$$\text{SNR} \propto \frac{\delta^2 P}{B}$$

- for pulsed case $B \sim 10^8$
- for CW case $B \sim 10$
- **large enhancement in SNR!**

For the CW case we lose temporal resolution; can not separate multiple modes or reflections from defects; complex standing wave pattern may be formed due to reflections from edges

- to overcome this problem, we scan the excitation frequency and reconstruct the “pulsed” response

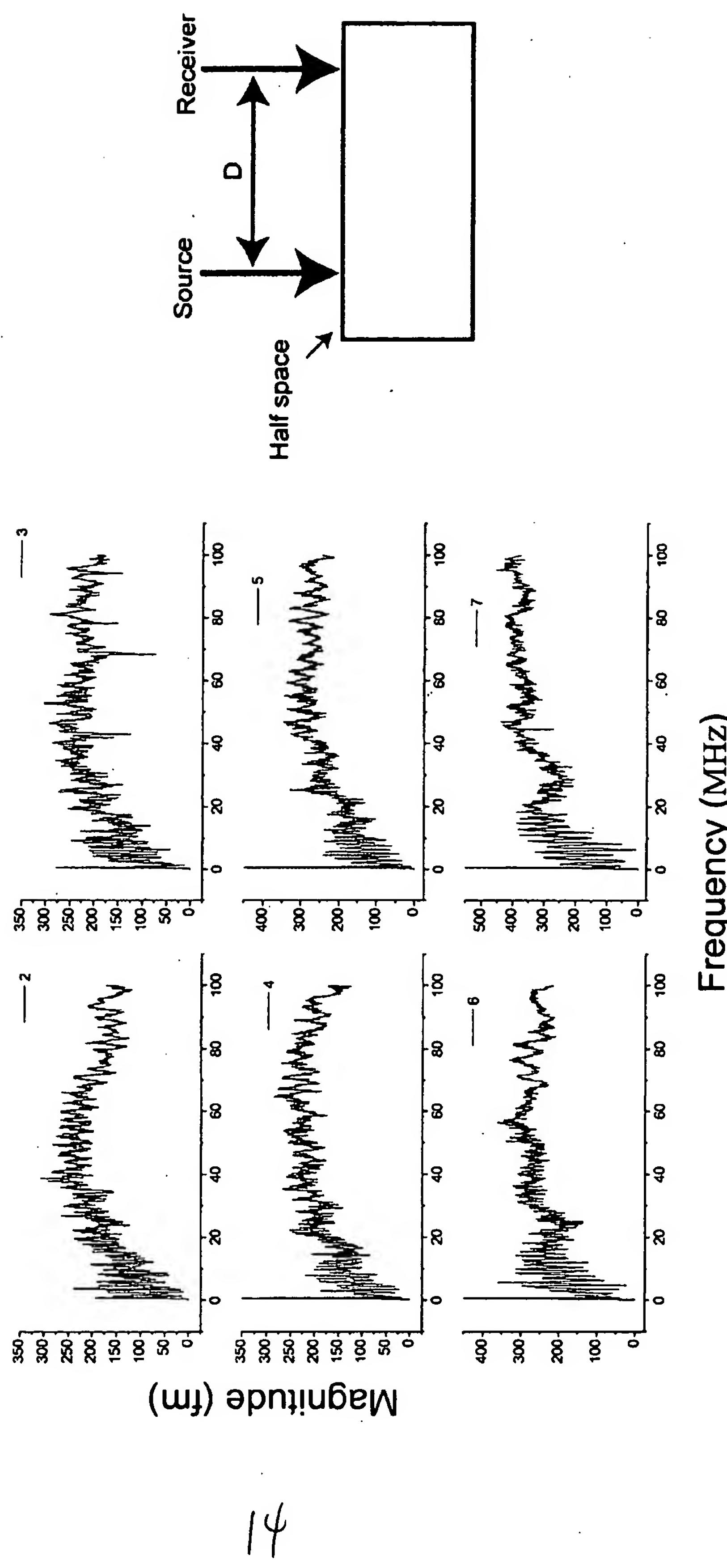
Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials



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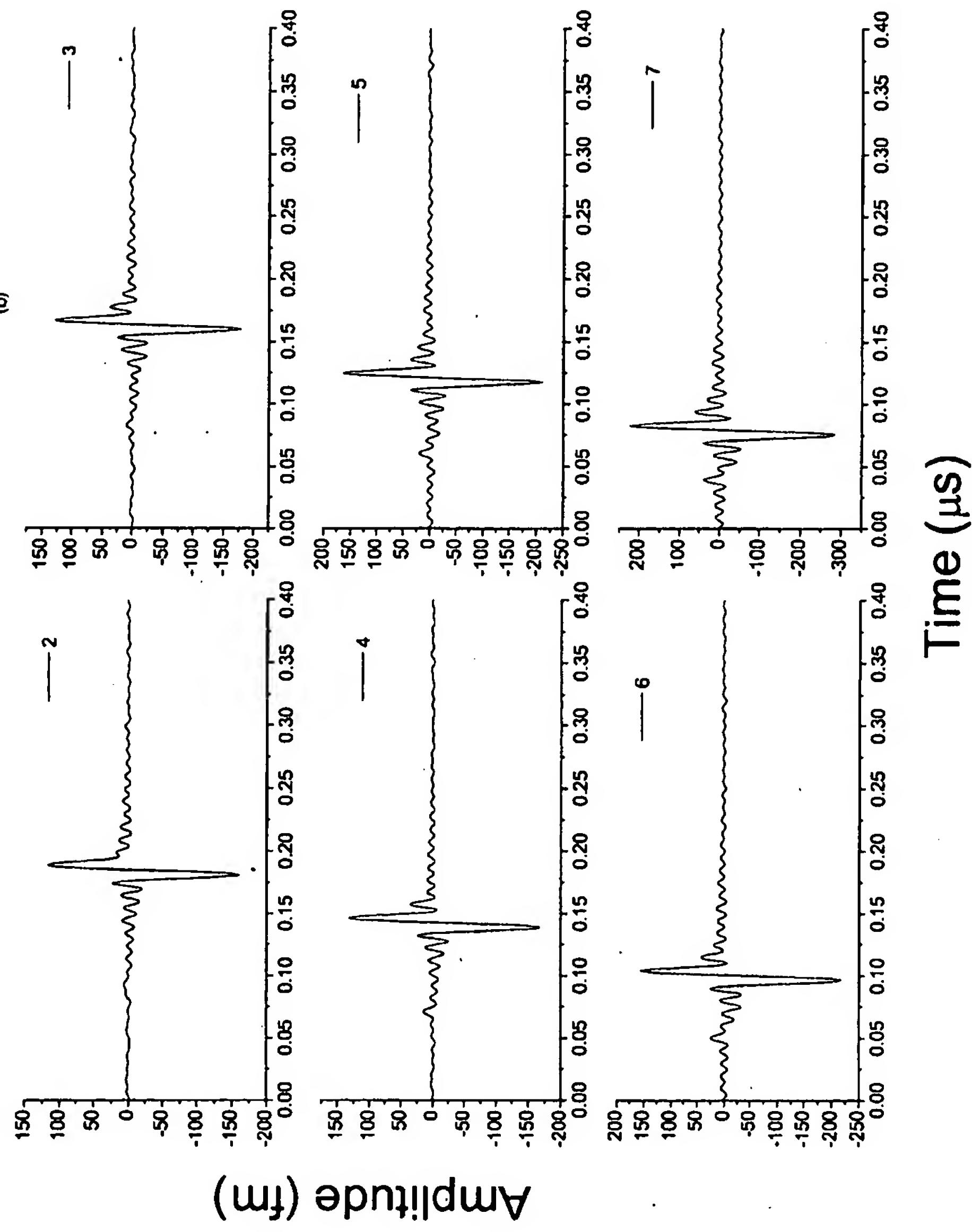
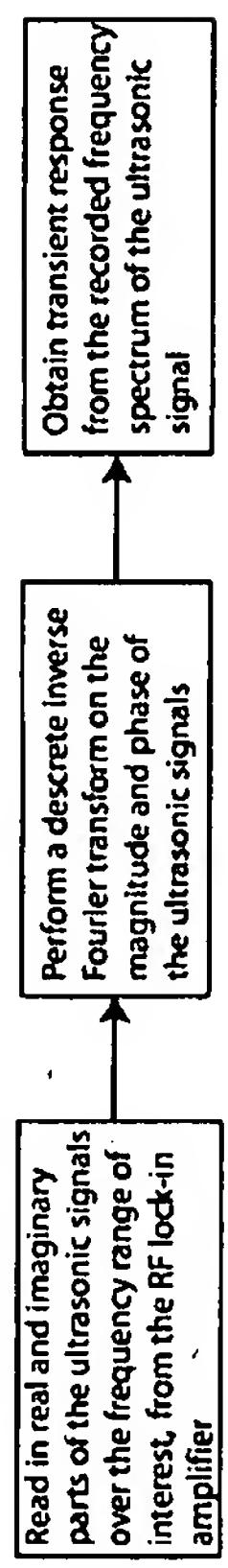
Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

Experimental Results: Frequency Domain Data on Aluminum Halfspace



Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials: Experimental Results: Reconstructed Time Domain Data, All Half Space

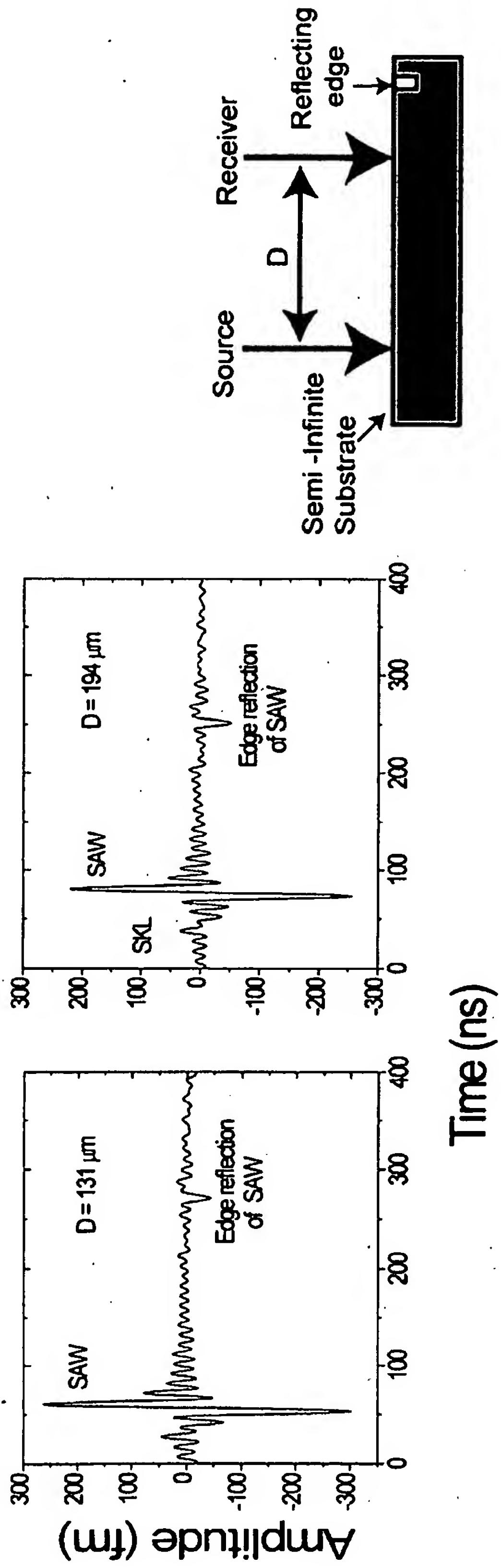
□ Signal processing scheme - Separation of modes in the time domain



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Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

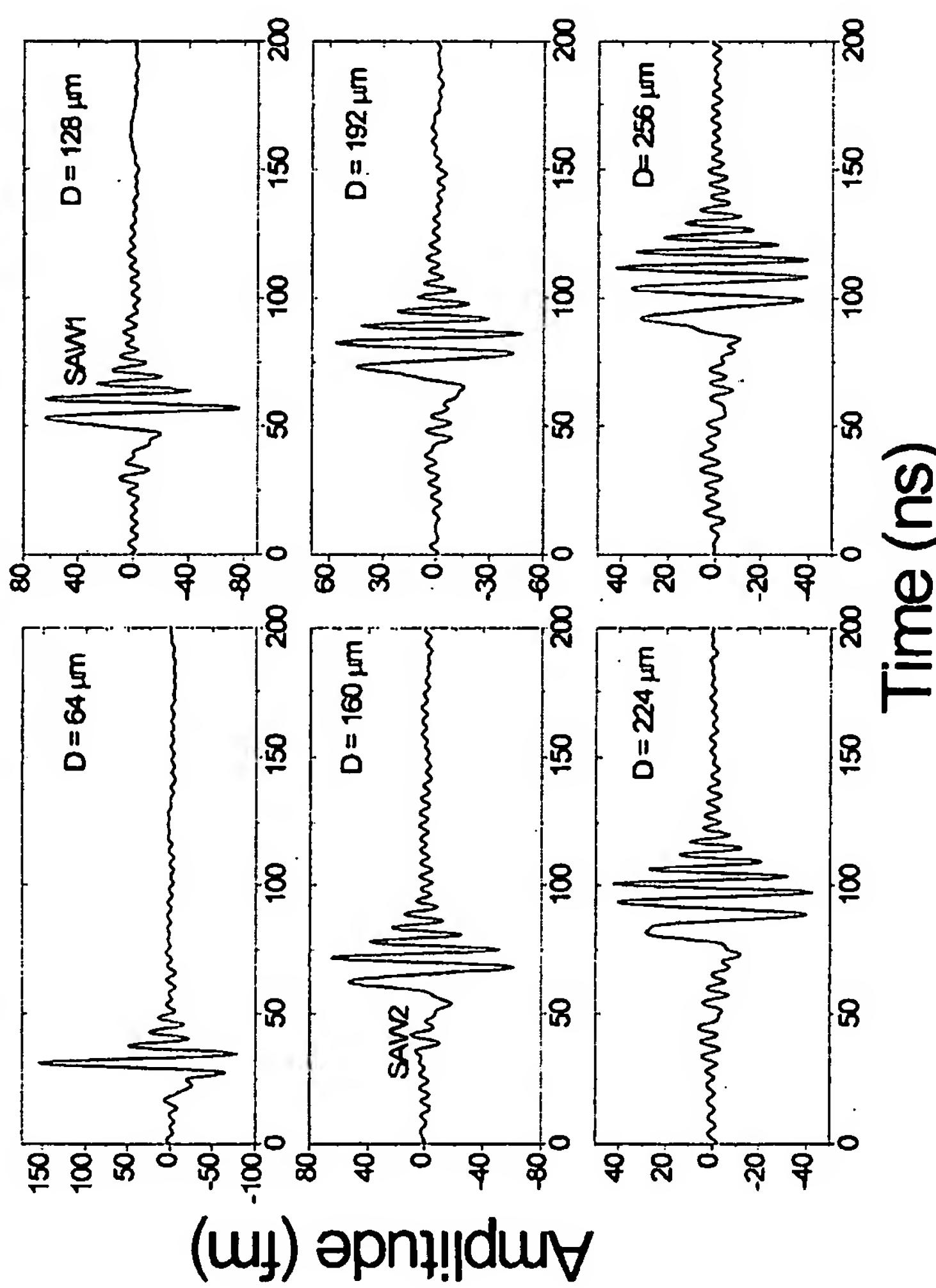
Reconstruction of time domain data allows us distinguish/ time gate individual arrivals in the signal



(a) Surface acoustic waves detected on a 6 mm thick aluminium block substrate as a function of source position. Each waveform was reconstructed from frequency domain data taken over the range 100 KHz to 200 MHz. (b) Surface acoustic waves reconstructed over the frequency range 100 KHz to 100 MHz. Frequency spectrum contains an edge reflection as seen in the time domain data. The bandwidth of the optical detection system at each frequency is 10Hz. SAW - Surface acoustic wave, SKL - Surface skimming longitudinal wave.

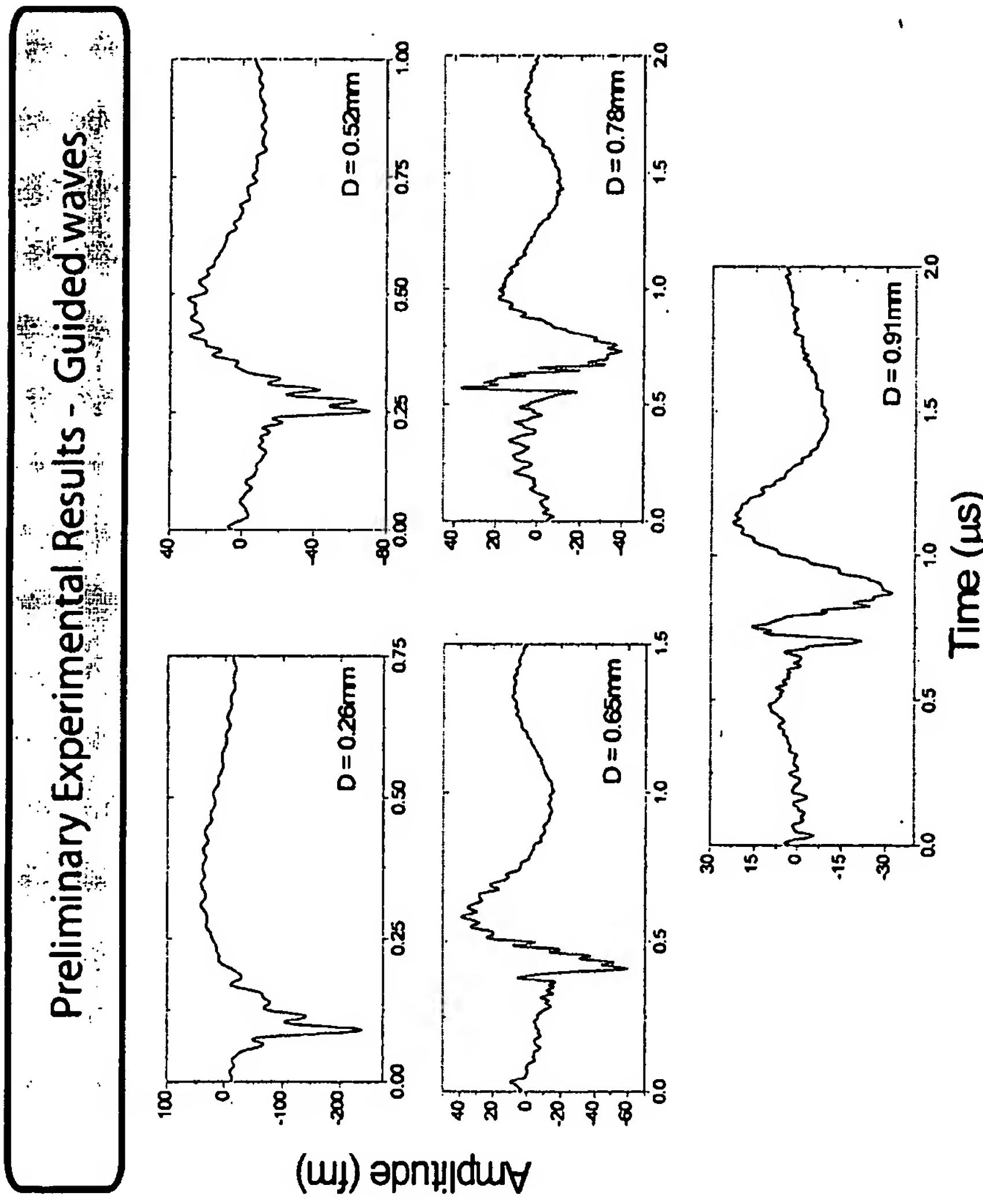
Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

Preliminary Experimental Results - Surface Waves in film/substrate system



Dispersive surface acoustic waves detected on a 240 nm gold film on a fused silica substrate as a function of source position. Each waveform was reconstructed from frequency domain data taken over the range 100 KHz to 200 MHz. The bandwidth of the optical detection system at each frequency is 10Hz. SAW1 - first surface acoustic wave mode. SAW2 - second surface acoustic wave mode.

Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials



Antisymmetric Lamb waves (A0 mode) detected on a 100 microns thick free standing aluminium plate.
Each waveform was reconstructed from frequency domain data taken over the range 100 KHz to 50 MHz.
The bandwidth of the optical detection system at each frequency is 10Hz.

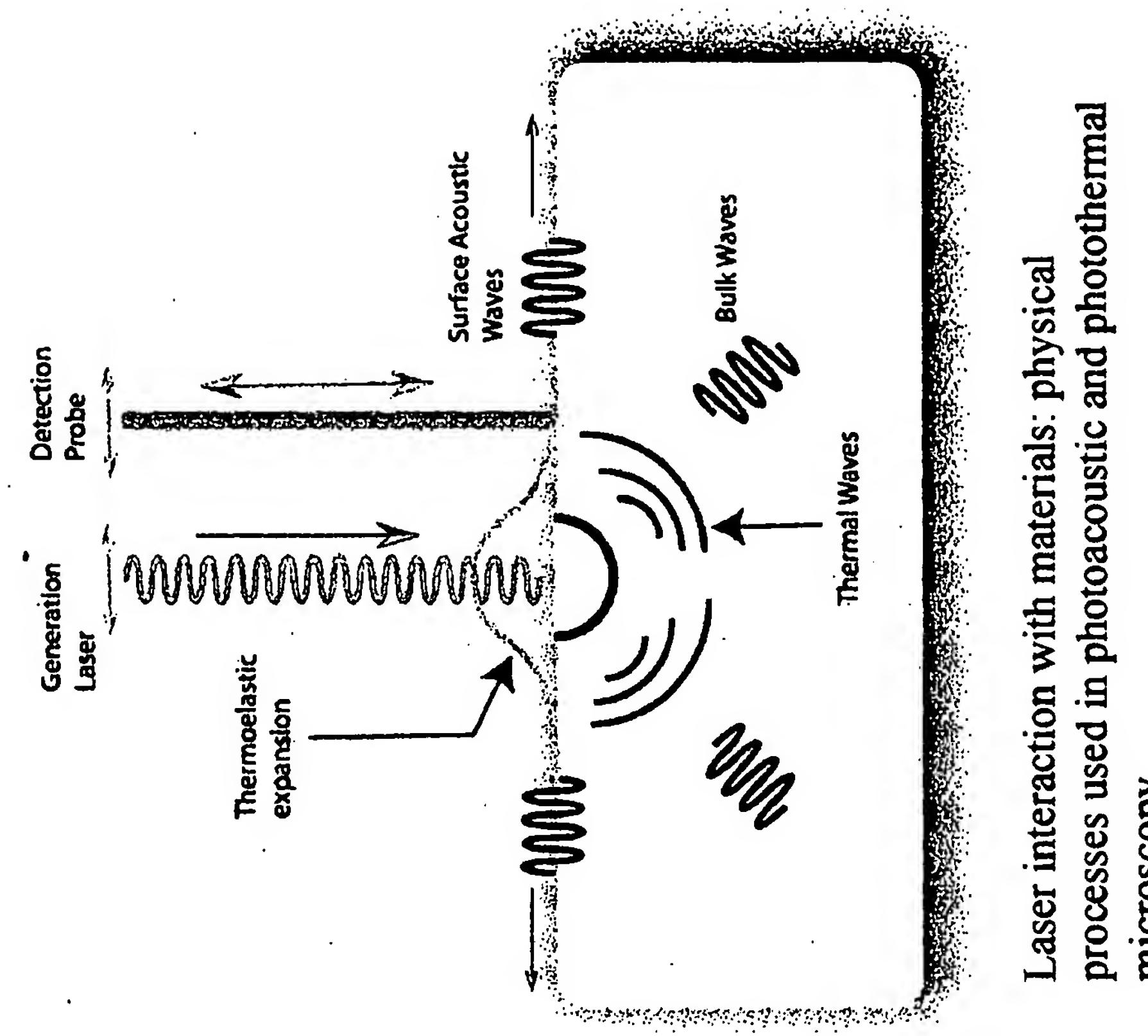
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Lower cost compared to broadband laser acoustic systems.

- ❑ Challenges
- ❑ CW generated ultrasonic signals may contain multiple modes that can render signal interpretation difficult.

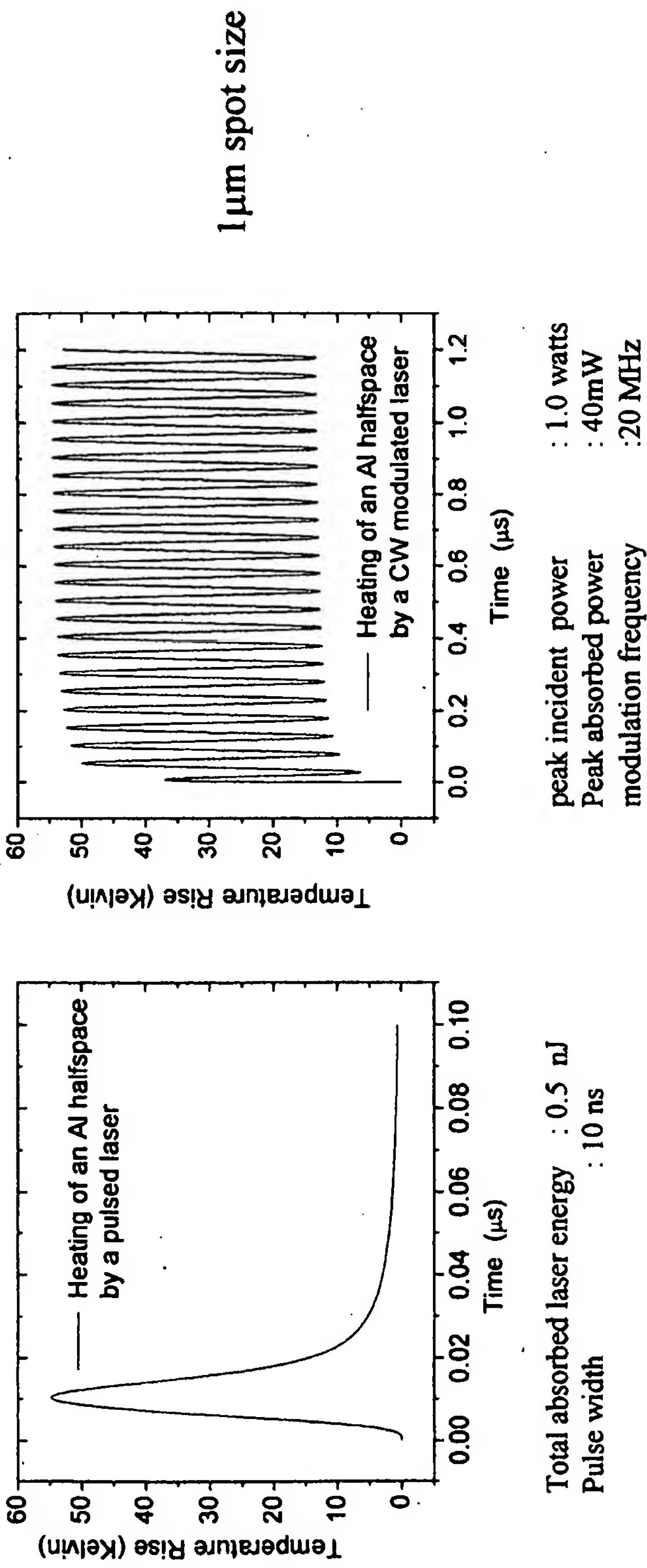
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Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

SNR of shot noise limited optical detection system:

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1. Multi-Pulse Methods 1,2

-use multiple laser sources to generate narrow bandwidth or phased acoustic pulses

2. Single Pulse Methods 3

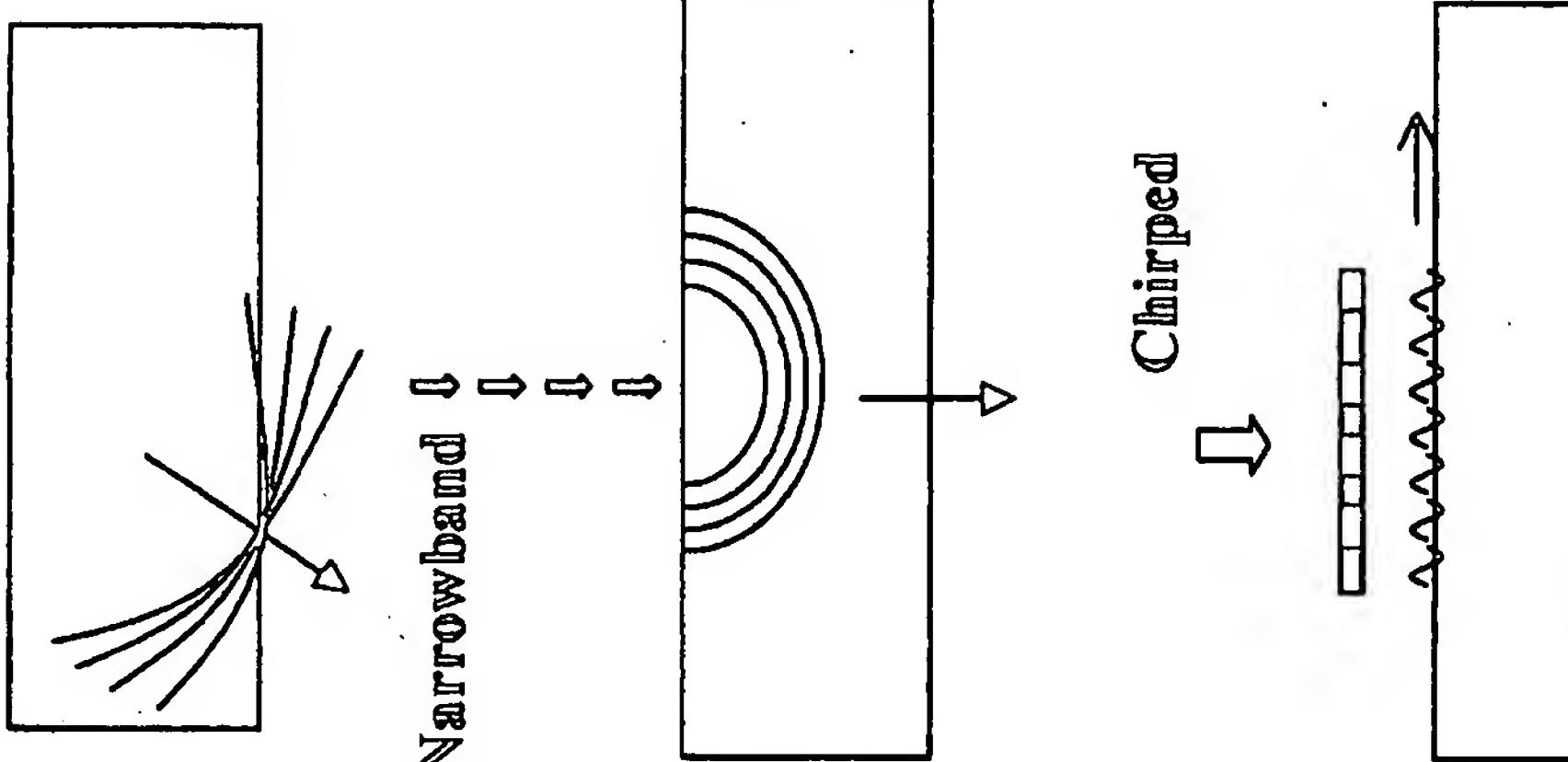
-modify spatial profile of single laser pulse to optimize SNR (narrow-band/ chirped)

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Phased array

\downarrow



1. T.W. Murray, J.B. Deaton Jr., and J.W. Wagner, "Experimental evaluation of enhanced generation of acoustic waves using an array of laser sources," *Ultrasonics* (1996)
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Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

Basic idea of sensitivity enhancement through CW generation

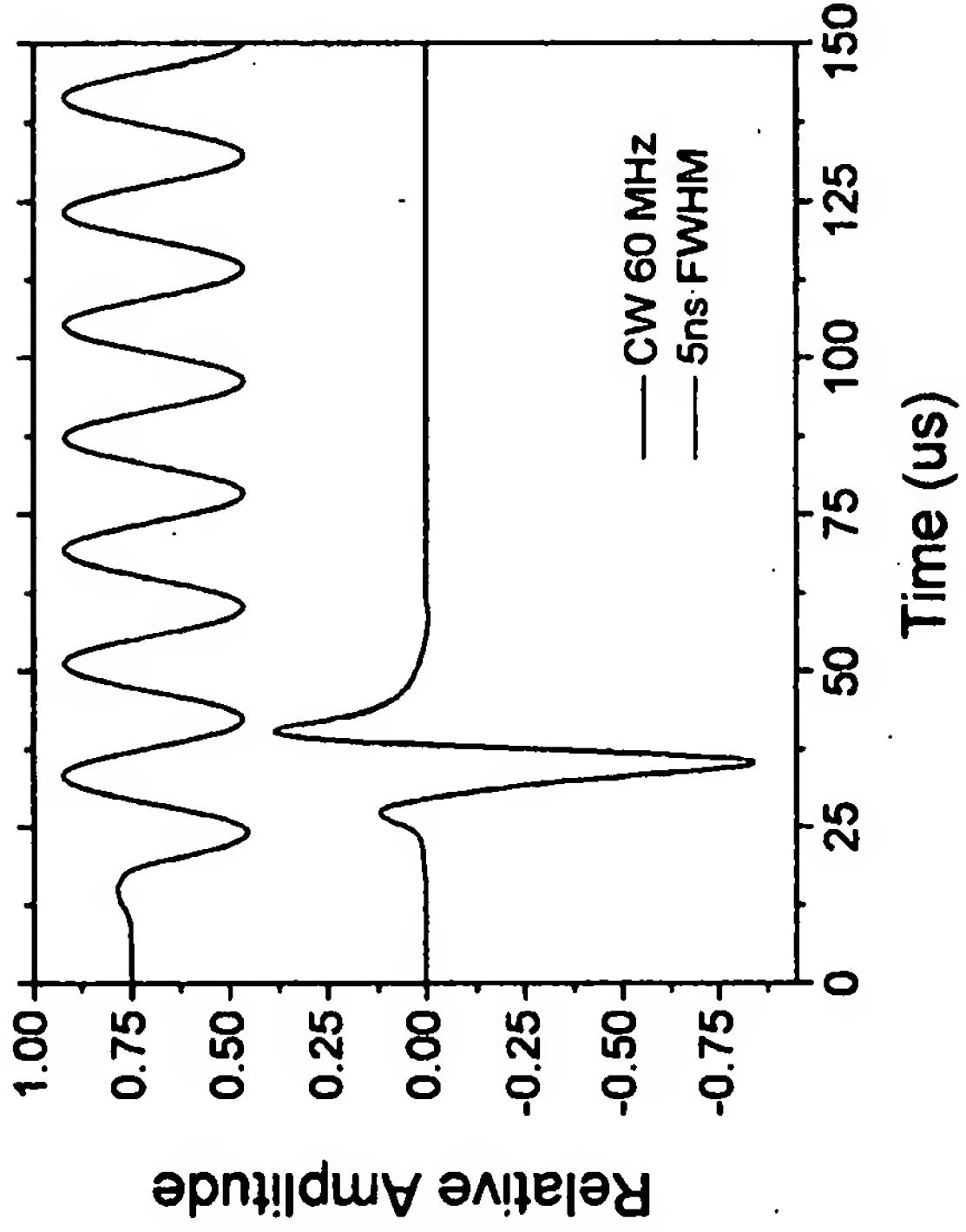
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All laser ultrasonic systems ultimately limited by

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35



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detection system:

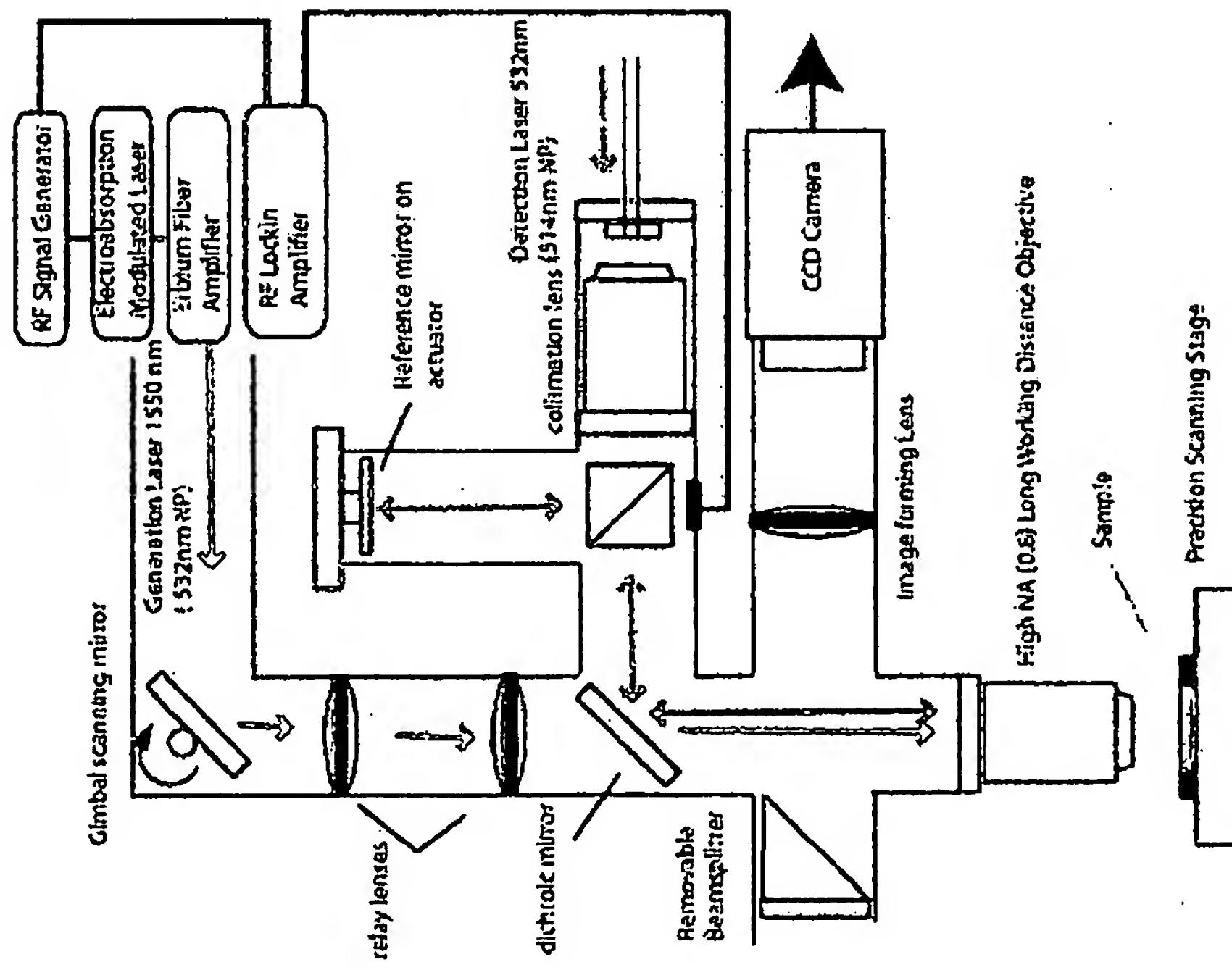
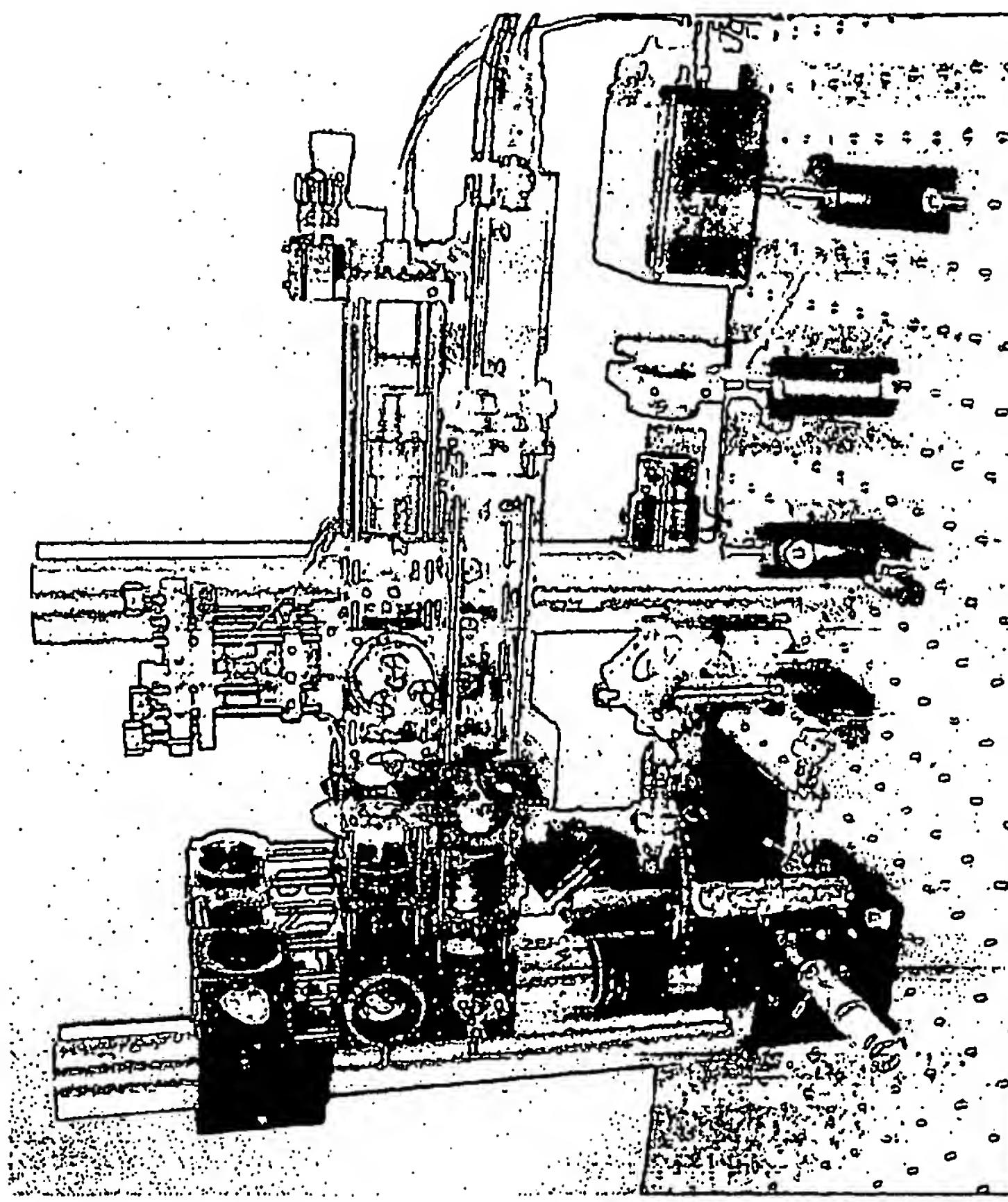
$$\text{SNR} \propto \frac{\delta^2 P}{B}$$

- for pulsed case $B \sim 10^8$
- for CW case $B \sim 10$
- large enhancement in SNR!

For the CW case we lose temporal resolution; can not separate multiple modes or reflections from defects; complex standing wave pattern may be formed due to reflections from edges

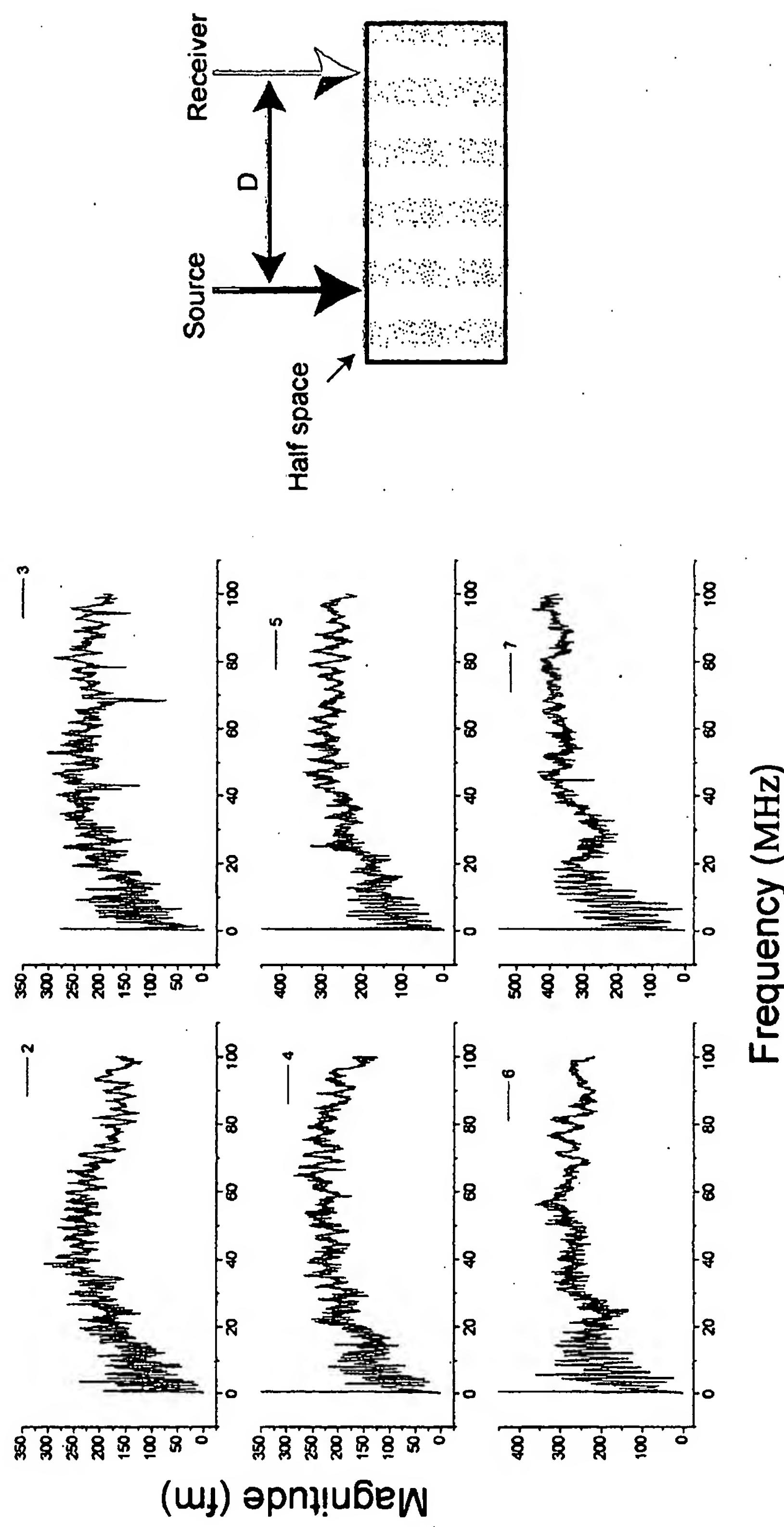
- to overcome this problem, we scan the excitation frequency and reconstruct the “pulsed” response

Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials



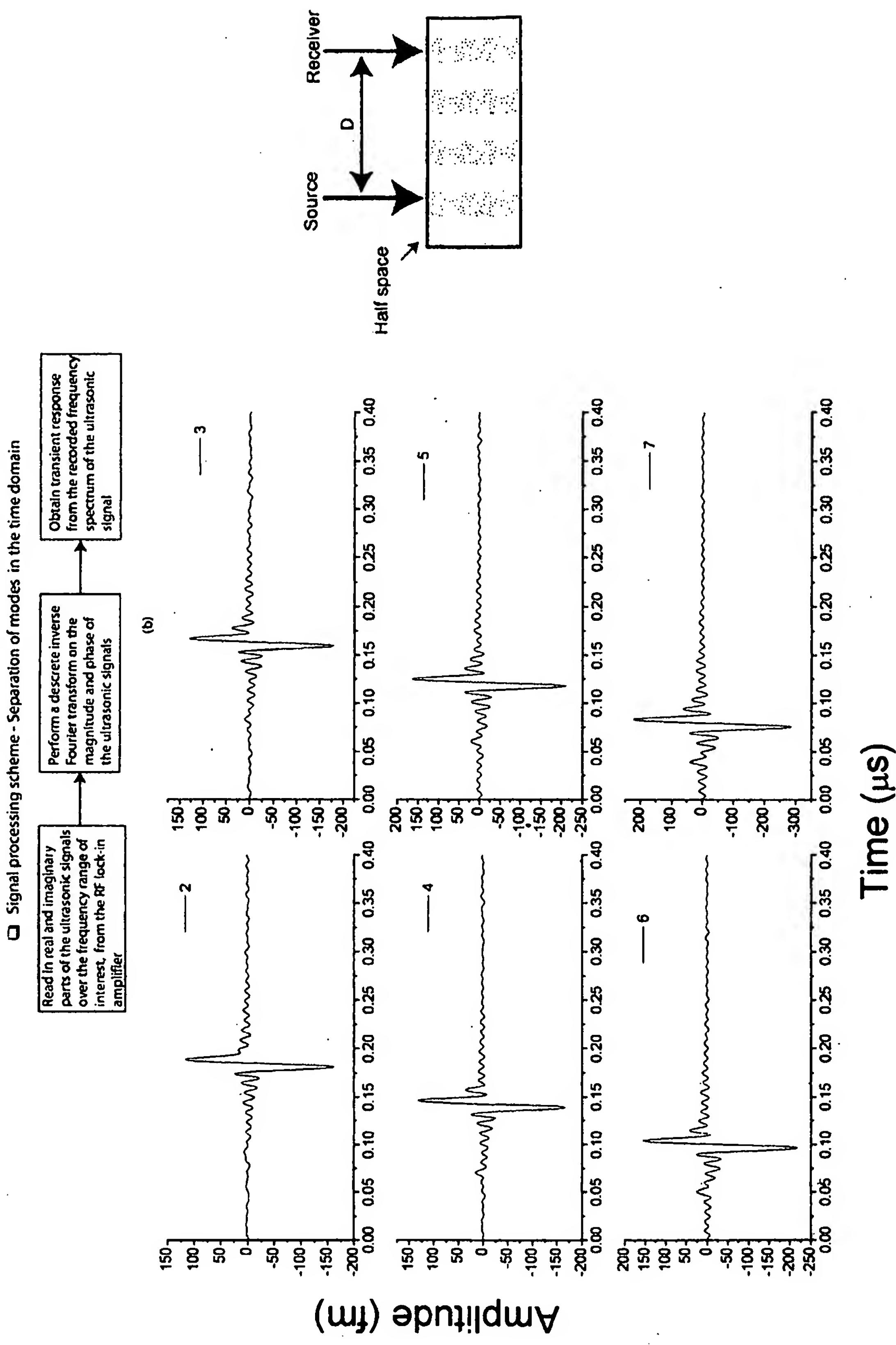
Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

Experimental Results: Frequency Domain Data on Aluminum Halfspace



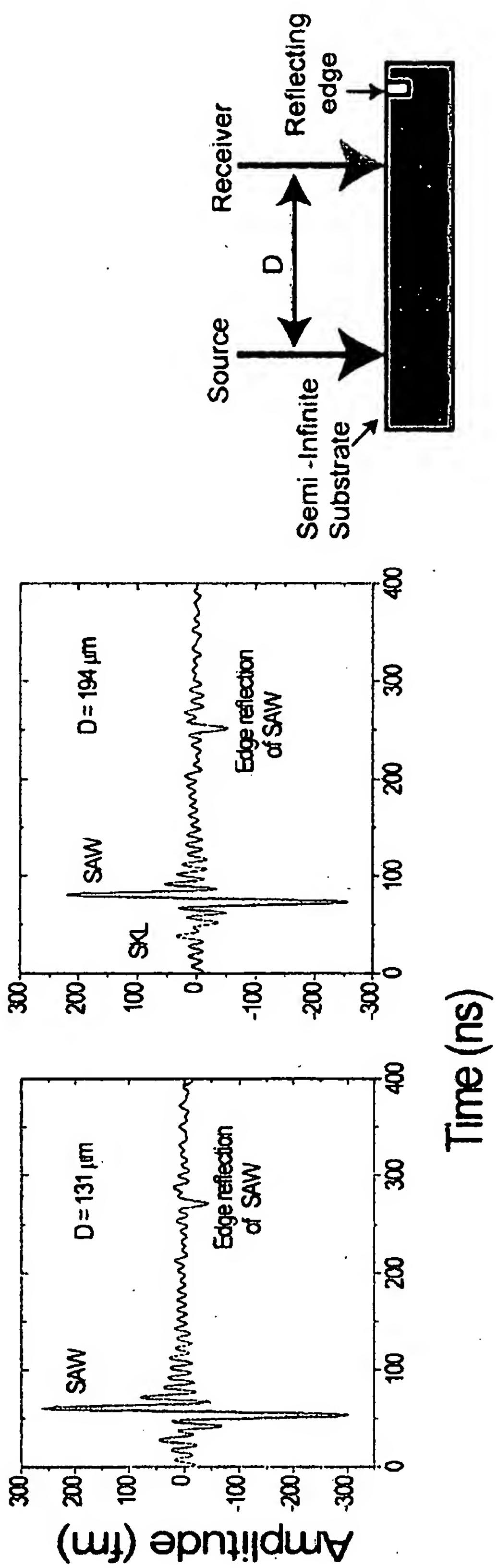
25

Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials: Experimental Results: Reconstructed Time Domain Data, Al Half Space



Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

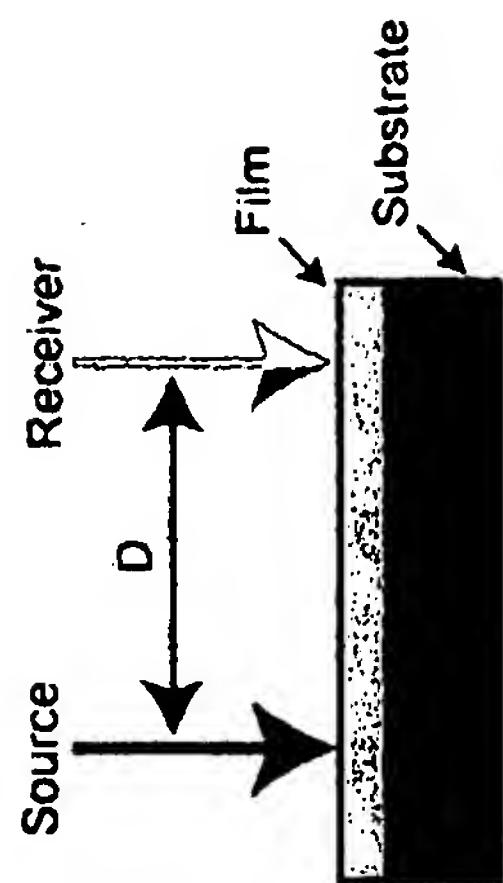
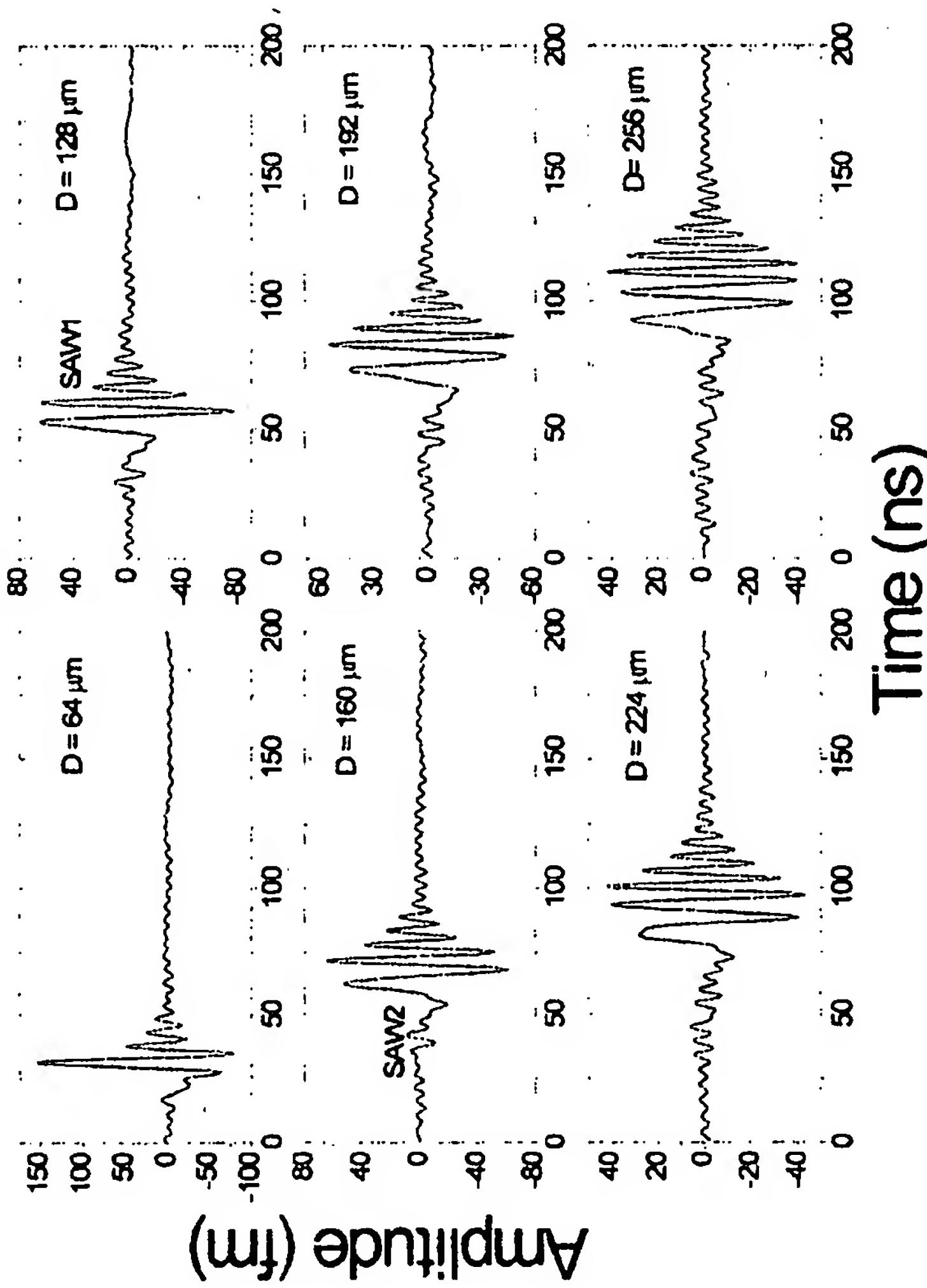
Reconstruction of time domain data allows us distinguish/ time gate individual (b) arrivals in the signal



(a) Surface acoustic waves detected on a 6 mm thick aluminium block substrate as a function of source position. Each waveform was reconstructed from frequency domain data taken over the range 100 KHz to 200 MHz. (b) Surface acoustic waves reconstructed over the frequency range 100 KHz to 100 MHz. Frequency spectrum contains an edge reflection as seen in the time domain data. The bandwidth of the optical detection system at each frequency is 10Hz. SAW - Surface acoustic wave, SKL - Surface skimming longitudinal wave.

Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

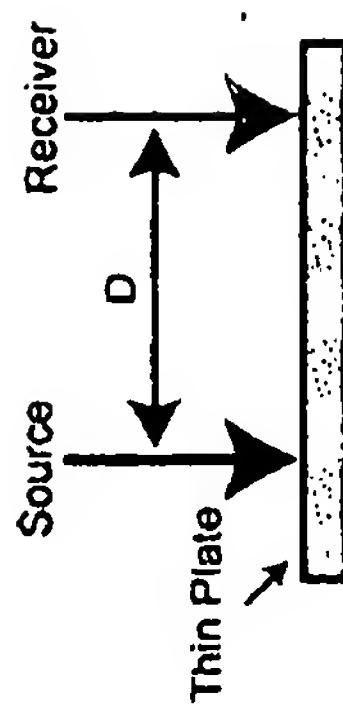
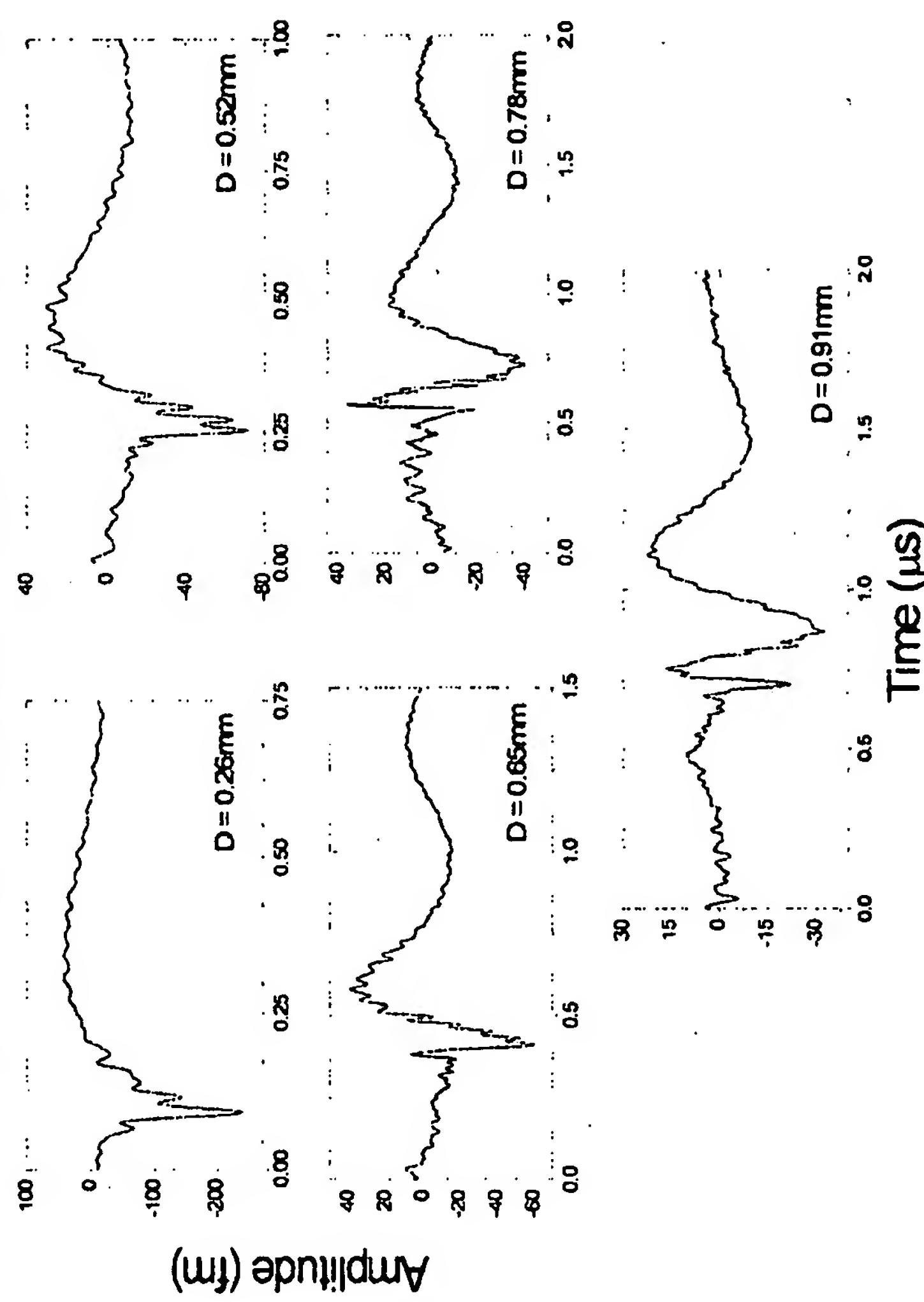
Preliminary Experimental Results - Surface Waves in film/substrate system



Dispersive surface acoustic waves detected on a 240 nm gold film on a fused silica substrate as a function of source position. Each waveform was reconstructed from frequency domain data taken over the range 100 KHz to 200 MHz. The bandwidth of the optical detection system at each frequency is 10Hz. SAW1 - first surface acoustic wave mode. SAW2 - second surface acoustic wave mode.

Device and Method for High Sensitivity Laser Ultrasonic Characterization of Micro and Nanoscale Materials

Preliminary Experimental Results - Guided waves



Antisymmetric Lamb waves (A0 mode) detected on a 100 microns thick free standing aluminium plate.
Each waveform was reconstructed from frequency domain data taken over the range 100 KHz to 50 MHz.
The bandwidth of the optical detection system at each frequency is 10Hz.